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THE RELATIONSHIP OF THERMAL  
STABILITY WITH STRUCTURE OF ORGANIC  
HIGH EXPLOSIVES, I. TRINITROPHENYL  
DERIVATIVES WITH AN ALPHA C-H  
LINKAGE (C)

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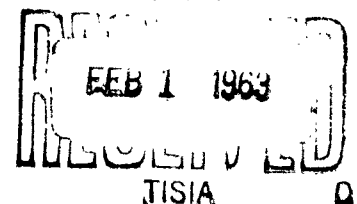
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The Relationship of Thermal Stability  
with Structure of Organic High Explosives, I.  
Trinitrophenyl Derivatives with an Alpha C-H Linkage (C)

Prepared by: Jerome M. Rosen  
Joseph C. Dacons

ABSTRACT: Trinitrobenzene has been used as a solvent to evaluate the thermal stability of solutions of seven structurally related compounds, each containing a symmetrical trinitrophenyl nucleus along with a substituent containing the alpha C-H linkage. Large differences in thermal stability exist and are accounted for by the ease of oxidative attack of the substituent by the nitro group. Compounds with easily oxidizable groups are the least stable. Thermal stability measurements of hexanitrostilbene in the solid and liquid states show clearly the extent to which this explosive is stabilized by its crystal environment.

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The recognition of the correlation of thermal reactivity, impact sensitivity and molecular structure in organic explosives has led to remarkable progress in the discovery of heat resistant explosives with a wide spectrum of sensitivities. The work reported here, performed under Task RUME 3E000/212 1/F008 10 004 017, Desensitization of Explosives, is a contribution to this general area.

R. E. ODENING  
Captain, USN

*Albert Lightbody*  
ALBERT LIGHTBODY  
By direction

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## INTRODUCTION

In the conventional vacuum stability test the effects of molecular structure cannot normally be distinguished from crystal lattice effects when a compound is solid at the test temperature. In the solid state the atoms involved in the thermal decomposition reaction are relatively immobile and thus it is difficult for chemical interactions to take place. By carrying out thermal stability measurements on solutions of the materials being tested in a nonreactive solvent, effects associated with the crystal lattice are eliminated and the stability becomes solely a function of molecular structure. This might be referred to as the intrinsic stability of the molecule.

Using this technique, valid comparisons of thermal stability may be made at test temperatures where certain of the compounds would normally be in the solid state and others normally in the melt. In the present report such comparisons are made within a series of structurally related compounds, each containing the symmetrical trinitrophenyl nucleus in addition to one or more substituent functional groups, the purpose being to determine the influence of the substituent group on thermal stability. This study is a logical extension of the TNT thermal decomposition study (1).

Although no relationship between thermal stability and structure of a related series of compounds has been previously demonstrated, a number of stability studies have been carried out on liquid explosives as well as explosives in solution. Farmer (2) observed the large difference between the rate of thermal decomposition of liquid and solid tetryl. He also found that the initial decomposition of solid tetryl was autocatalytic and ascribed this to progressive melting. Further work by Hinshelwood (3) demonstrated that the acceleration of the decomposition of solid tetryl could be quantitatively accounted for by progressive melting.

Henkin (4) showed clearly the effects of liquefaction on the stability of tetramethylcyclohexanol pentanitrate (Sixolite), N,N'-dinitrodinimethyloxamide (MNO) and N,N'-dinitrodi( $\beta$ -nitroxyethyl)oxamide (NENO) by stability measurements of the explosives dissolved in TNT. He also compared the rate of decomposition of liquid (or dissolved) explosive with that of the solid. Similar comparisons have been made for PETN (5) and bis(trinitroethyl)nitramine, BTNEN (6).



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Ratios of the initial rates of decomposition of liquid and solid explosives are given in Table I. In each case shown in Table I the ratio of liquid to solid decomposition is determined below the melting point. Measurements of rate of liquid decomposition below the melting point are made from solution using a nonreactive solvent.

DISCUSSION AND RESULTS

1,3,5-Trinitrobenzene, TNB, has been found to be an excellent and reasonably thermally stable solvent for measurement of the thermal decomposition of dissolved trinitrophenyl compounds. A purified sample, heated for three hours at 300°C, produced less than 0.5 cc of gas per gram. The melting point of this heated material did not differ significantly from that of an unheated control. Blais also has commented (8) on the excellent thermal stability of TNB. The major difficulty in using TNB in the present study results from its high vapor pressure at elevated temperatures (9).

The recent study of the thermal decomposition of TNT (1) has shown quite clearly that the preferred decomposition process involves an oxidative attack at the methyl group by the nitro group, this probably taking place by both inter- and intramolecular reactions. The unusual thermal stability of TNB is now apparent, as there is no easy access for oxidative attack.

Kamlet and Sickman (10), in relating impact sensitivity to molecular structure, found that trinitrophenyl compounds containing an alpha carbon-hydrogen linkage appeared to comprise a distinct sensitivity class in which there existed a linear relationship between the logarithmic 50% impact height and the "oxidant balance." They suggested that for this class of compounds the initial decomposition was similar to that in TNT, taking place preferentially at the alpha carbon-hydrogen position. It seemed to us worthwhile to examine several members of this same class from the standpoint of the thermal stability. Vacuum stability data on trinitrobenzene solutions of seven trinitrophenyl compounds, each with an alpha C-H linkage, are assembled in Table II.

Since the compounds studied in this work all contain the alpha C-H linkage, there is reason to believe they undergo similar initial modes of decomposition. It is assumed that initial gas evolution measurements will serve to place these compounds in the proper order of thermal stability.

Carboxaldehydes and carbinols are classically more susceptible to chemical oxidation than unsubstituted methyl groups. It is therefore not surprising that trinitrobenzaldehyde proved to be the least stable member of the group, followed by trinitrobenzyl alcohol. Both of these compounds are more sensitive than would be predicted from the impact sensitivity-oxidant balance relationship (10). Trinitrobenzaldehyde and trinitrobenzyl alcohol decompose readily at 200°C, whereas in the case of TNT, it is necessary to heat to 230°C to get a measurable quantity of gas.

Hexanitrobibenzyl, which may be viewed as a dimer of TNT showed about two and one half times the thermal decomposition rate of TNT at the same temperature. Although the decomposition routes are not necessarily the same, the relatively close correspondence of the rates implies a similarity in the rate determining steps.

The decomposition of trinitro-m-cresol appears to be more strongly influenced by the hydroxyl than by the methyl group, as its decomposition rate, Table II, corresponds more closely to that of picric acid than to TNT. It is interesting to note that trinitro-m-cresol does not act as expected at temperatures generated under the impact hammer as it is much less sensitive than a TNT derivative, as pointed out by Kamlet and Sickman (10).

The somewhat greater thermal stability of hexanitrostilbene as compared with 2,4,6-trinitrostilbene was not anticipated, but perhaps can be rationalized. An electron-rich carbon atom is more easily oxidized than one which is electron-deficient. The carbon atom alpha to the nitrated ring in trinitrostilbene and those alpha to either ring in hexanitrostilbene are electron deficient relative to the carbon alpha to the unnitrated ring of trinitrostilbene as a result of the electron withdrawing character of the nitro groups. Thus the carbon atom alpha to the unnitrated ring would be more reactive in an oxidation-reduction type reaction. The presence of this more reactive site in trinitrostilbene may contribute to its greater rate of decomposition relative to hexanitrostilbene. Splitter and Calvin (11) have reported on the accelerating effect of a 4'-dimethylamino group on the photochemical oxidative rearrangement of 2,4,6-trinitrostilbene. They have suggested that the reaction involves the interaction of a nitro group with an alpha carbon atom which is not too electron-deficient.

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Decomposition rates of a number of the compounds were measured at more than one temperature, Table III. No attempts were made to calculate activation energies from these data as the technique cannot be extended to cover a sufficiently wide temperature range, nor were the measurements of sufficient accuracy to warrant this. However, it seems that the temperature coefficient of the decomposition rates fall within normal ranges.

A few measurements were made at other than 5 mole percent concentration, Table IV. The absence of a marked concentration effect was not completely unexpected, nor is it inconsistent with the isolation of products in the TNT thermal decomposition study (1) which could have resulted only from bimolecular reactions.

The demanding requirements of a solvent for this present study - high heat stability, resistance to oxidation and reduction type reactions, and relatively low vapor pressure - made it difficult to select suitable solvents. For the most part, trinitrobenzene met these requirements. Its excellent thermal stability has been demonstrated. Although TNT has a somewhat lower thermal stability than TNB, Robertson (12) successfully employed TNT as a solvent in a thermal stability study of RDX at temperatures up to 281°C. The very large difference in rate of decomposition of RDX compared to TNT made this possible.

The question of whether there is a solute-solvent interaction is not easy to answer. There would appear to be no reason to believe that the nitro groups of TNB are more reactive than the nitro groups of the other compounds studied. A polarographic study lends some support as it is shown there is no significant difference in reduction potential between TNT and TNB in the first step reduction (13).

In an attempt to answer the question of a solute-solvent interaction, diphenylsulfone was selected for comparison with TNB as it appeared to meet the solvent requirements stated above. Although diphenylsulfone shows no decomposition at 260°C gas evolution measurements at 210°C with dissolved trinitro-m-cresol showed a somewhat greater rate of decomposition than this same compound dissolved in TNB, Figure 1. A rapidly rising rate of decomposition is obtained with TNT in diphenylsulfone at 230°C, while a nearly steady rate is obtained in TNB at this same temperature. It is evident that diphenylsulfone is not a suitable solvent for our work. Although the possibility of a

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TNB interaction has not been ruled out, it is suggested that it is minimal and that the solvent does not alter the thermal stability ordering in Table II.

Thermal stability measurements of hexanitrostilbene in the solid and liquid states have been made. The very great difference in stability between the solid and liquid, Table V, shows clearly the extent to which the crystal environment stabilizes this explosive. It is easy to visualize how a small amount of impurity could degrade its stability, as the addition of a substance in which hexanitrostilbene is soluble would cause a considerable increase in the rate of decomposition. Certainly hexanitrostilbene is a good example of an explosive that possesses unusual thermal stability by virtue of strong crystal lattice forces.

EXPERIMENTAL

Gas evolution measurements were made by the vacuum stability method which used a sealed one-piece glass tube and monometer (14). The volume of each tube, which ranged from about 8 to 10 cc in volume, was determined by a calibration before use. In the calculations, an allowance was made for the volume occupied by the sample and solvent. Liquid trinitrobenzene was assumed to have a density of 1.4. Test temperatures were selected to obtain easily measurable initial rates of decomposition, generally falling within 1 and 5 cc of gas per gram of solute per hour. Values reported were those resulting from the initial decomposition and were assumed to have been derived from the solute only.

Measurements of the pressure increase resulting from decomposition were taken after sufficient time was allowed for the sample to dissolve and for the solvent to reach the test temperature. The minimum time allowed for this was thirty minutes. During this initial period a pressure rise was observed which resulted from a number of factors such as vapor pressure and expansion of the solvent. In addition, some of the measurements were started with about 100 mm of air at room temperature in the apparatus in order to reduce the rate of solvent diffusion into the capillary tubing. This proved helpful at temperatures of 230°C or higher, where trinitrobenzene had an appreciable vapor pressure.

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Very slowly increasing rates of gas evolution were obtained from 2,4,6-trinitrostilbene, hexanitrostilbene and hexanitrobibenzyl. TNT appeared to produce gas at a nearly steady rate at 230°C and a very slowly increasing rate at 240°C. Slowly decreasing rates were shown by picric acid, trinitro-m-cresol, trinitrobenzaldehyde and trinitrobenzyl alcohol.

In nearly every case 50 milligrams of a compound were used with the appropriate quantity of TNB. At the 5 mole percent level, 50 milligrams of hexanitrostilbene required 450 milligrams of TNB while 50 milligrams of trinitro-m-cresol required 834 milligrams of TNB.

Single measurements were made in most cases. It was more difficult to obtain good data at the higher temperatures, largely because of the volatility of the TNB. Temperature control was important, particularly when starting with a partial vacuum. The constant temperature equipment was maintained at  $\pm 0.3^\circ\text{C}$ . Another possible source of error was small amounts of volatiles remaining in the trinitrobenzene, although it was dried thoroughly. It is believed the values reported are reliable to  $\pm 0.3$  cc; however, insufficient measurements were made to obtain a good estimate of reproducibility.

All of the compounds studied, Table VI, were purified by recrystallization and dried thoroughly to remove residual solvent.

#### ACKNOWLEDGEMENTS

The authors are pleased to acknowledge the assistance of Mr. Herbert T. Simmons, Jr. and the helpful suggestions of Drs. M. J. Kamlet and D. V. Sickman. A part of the compounds studied were prepared by Dr. Kathryn G. Shipp.

TABLE I

Ratios of Initial Rates of Decomposition  
of Liquid ( $K_L$ ) and Solid ( $K_S$ ) Explosives

<u>Compound</u>	<u>Ratio, <math>\frac{K_L}{K_S}</math></u>	<u>Temp., °C</u>	<u>Reference</u>
BTNEN	50	90	(6)
Tetryl	30 60	100 120	(7)
PETN	20	120	(5)
MNO	70	100	(4)
NENO	20	85	(4)
Sixolite	50	100	(4)

TABLE II

Initial Decomposition Rates of Five  
Mole Percent Solutions in Trinitrobenzene

<u>Compound</u>	<u>Temp., °C</u>	<u>cc gas at NTP/ g/hour    <u>/a</u></u>
2,4,6-Trinitrobenzaldehyde	200	27
2,4,6-Trinitrobenzyl alcohol	200	3.2
2,4,6-Trinitro-m-cresol	210	7.0;7.1
2,4,6-Trinitrostilbene	210	1.3
2,2',4,4',6,6'-Hexanitrostilbene	230	1.6;1.8;1.9;2.0
2,2',4,4',6,6'-Hexanitrobibenzyl	230	1.6
2,4,6-Trinitrotoluene	230	0.6;0.6
(Picric acid)	210	2.8

---

/a The gas evolved is based on one gram of the compound listed and does not consider the amount of solvent.

TABLE III

Decomposition Rates in Trinitrobenzene  
as a Function of Temperature

<u>Compound</u>	<u>Temp., °C</u>	<u>cc gas at NTP/ g/hour /a</u>
Trinitro-m-cresol 5 mole %	200	2.7
	210	7.0
	220	15.5
Trinitrostilbene 5 mole %	210	1.3
	220	3.9
	230	13.6
Hexanitrostilbene 5 mole %	230	1.8
	240	3.9
Hexanitrobibenzyl 5 mole %	230	1.6
	240	3.4
TNT 5 mole %	230	0.6
	240	1.4

---

/a The gas evolved is based on one gram of the compound listed and does not consider the amount of solvent.



TABLE IV

Decomposition Rates in Trinitrobenzene  
at 230°C

<u>Compound</u>	<u>Concentration, Mole Percent</u>	<u>cc gas at NTP/ g/hour <u>/a</u></u>
TNT	2	0.8
	5	0.6
Hexanitrobibenzyl	2	1.8
	5	1.6
	10	2.0

---

/a The gas evolved is based on one gram of the compound listed and does not consider the amount of solvent.

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TABLE V

Initial Rate of Decomposition of Solid  
and Liquid Hexanitrostilbene /a

<u>Temp., °C</u>	cc gas at NTP/g/hour		<u>Ratio</u>
	<u>Solid Phase</u>	<u>Liquid Phase</u> <u>/b</u>	
230	0.04	1.8	45
260	0.4	28	70
280	2.0	-	-

/a This explosive, prepared for the first time at  
this Laboratory, is the subject of a recent report(15).

/b 5 mole percent in trinitrobenzene.

TABLE VI

<u>Compound</u>	<u>Melting Point, °C</u>
2,4,6-Trinitrobenzaldehyde	118-119
2,4,6-Trinitrobenzyl alcohol	102-103
2,4,6-Trinitro-m-cresol	106
2,4,6-Trinitrostilbene	157-158
2,2',4,4',6,6'-Hexanitrostilbene	316
2,2',4,4',6,6'-Hexanitrobibenzyl	215-216
TNT	80.7-80.8
2,4,6-Trinitrophenol	122

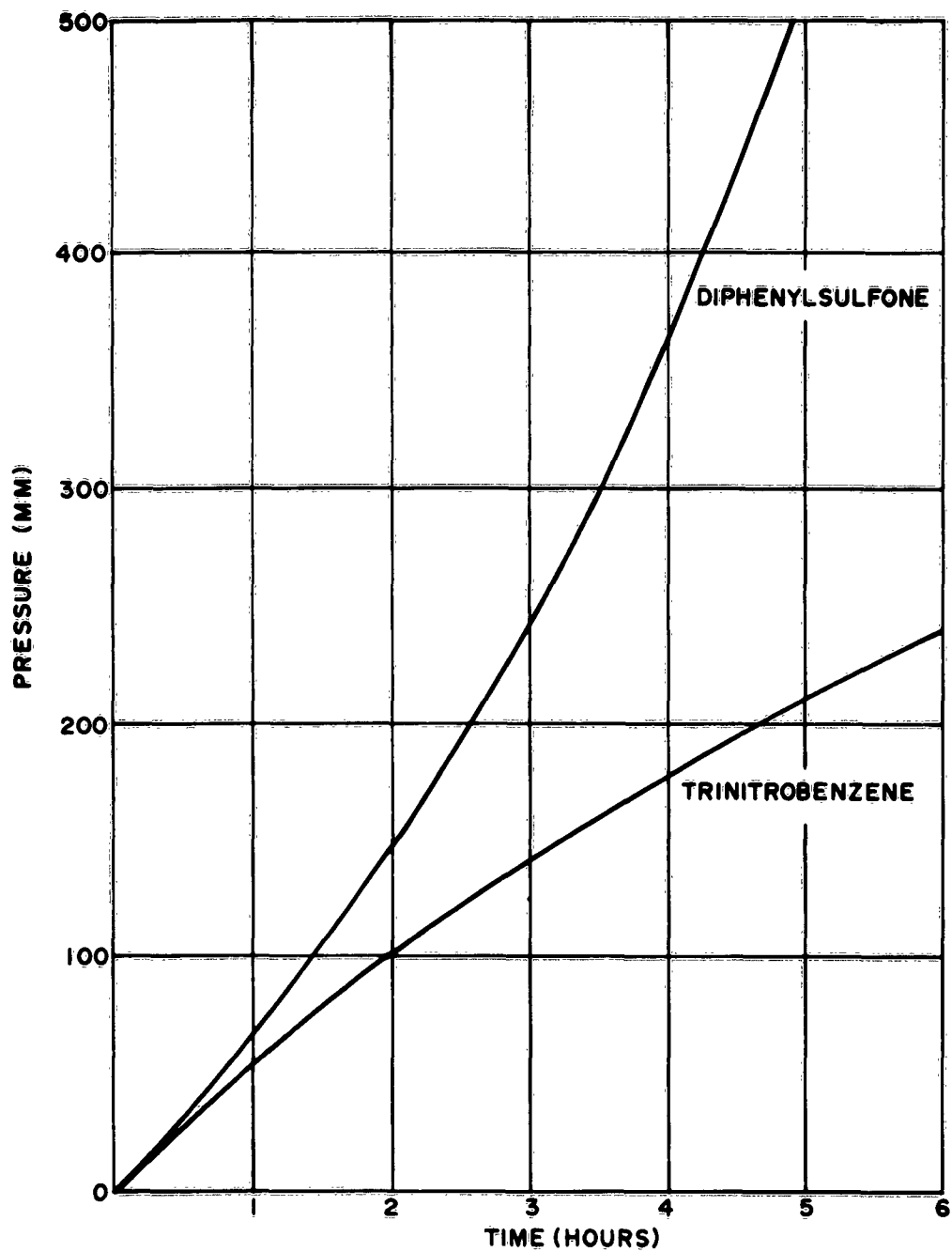


FIG. 1 THERMAL DECOMPOSITION OF 2,4,6 TRINITRO-m-CRESOL  
(5 MOLE %) IN DIPHENYLSULFONE AND TRINITROBENZENE  
AT 210°C

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Thermal	THER		Hexa	HEXA
Stability	STBI		Stilbene	STIL
Structure	STRC		Crystal	CRYS
Relationship	RELT		Environment	ENVI
Correlation	CORA			
Explosives	EXPL			
Organic	ORGA			
Trinitro	TRIT			
Phenyl	PHEN			
Derivatives	DERI			
Benzene	BENZ			



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(MOL technical report 62-192)  
THE RELATIONSHIP THERMAL STABILITY WITH  
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LINKAGE (C), by Jerome M. Rosen and Joseph C.  
Dacons. 15 Nov. 1962. 14p. tables. Task  
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1. Explosives,  
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  2. Explosives -  
Stability
  3. Trinitrophenyl
  4. Trinitroben-  
zene
  5. Hexanitrostil-  
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DOD DIR 5200.10.

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  2. Explosives -  
Stability
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  4. Trinitroben-  
zene
  5. Hexanitrostil-  
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  - I. Title
  - II. Rosen, Jerome M.
  - III. Dacons,  
Joseph C.,  
jt. author
  - IV. Project
- Abstract card  
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Naval Ordnance Laboratory, White Oak, Md.  
(MOL technical report 62-192)  
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